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ULTRAFINE-GRAINED SUPERCONDUCTORS. (U)

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ULTRAFINE-GRAINED SUPERCONDUCTORS

By

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1. Annual Report, Ultrafine-Grained Superconductors

January 1, 1981 to December 31, 1981

AFOSR Contract No. F49620-78-C-0031

M. Ashkin, A. I. Braginski and J. R. Gavaler

2. ABSTRACT

Measurements of the effect of strain on the critical current of NbN show that almost no degradation occurs even at the highest strain levels, 0.5%, and the highest fields, 22T, that could be applied. Data at 4.2K in pulsed fields of over 30T suggest that the enhanced critical field measured in ultrafine-grained NbN films are due to a dimensional effect. Data on laser annealed Nb-Ge films further elucidated the role of second-phase pinning in enhancing the critical currents in these films. Electron beam annealing did not enhance significantly the critical current density. Pseudobinary V-Si-C alloys with critical temperatures of 17K were prepared by C-implantation of vanadium-rich V-Si films and subsequent annealing. A theory of current distribution near the end of a filamentary conductor and the effect on ac losses was developed. Some features of the anomalous SAW attenuation measured in NbN films were explained using the Kosterlitz-Thouless theory.

3. OBJECTIVES

The original objectives of this program can be briefly summarized as follows:

1. To investigate the mechanism of critical-current density, J_c , enhancement in ultrafine-grained films of high T_c superconductors.
2. To investigate the preparation of ultrafine-grained microstructures of Al5 and B1 superconductors.
3. To determine the effect on T_c of variations in grain size in such Al5 and B1 superconductors.
4. To correlate mechanical load properties and microstructures.

In the course of this work, two additional narrower objectives have been formulated:

5. To investigate the stabilization of high- T_c Al5 and B1 phases during the film growth.
6. To experimentally investigate ac losses in "in-situ" formed multifilamentary Al5 composites.

High-temperature superconducting materials are needed for applications in airborne multimegawatt power generation and conditioning systems. Research directed toward the synthesis and characterization of high-transition temperature (T_c) compounds can eventually provide a greater margin of design efficiency and operational reliability than obtained with presently-available superconductors. To become technologically important, a superconductor must possess a high critical-current density (J_c) in addition to having a high T_c . In the present program research has been focussed on the investigation of the effect on J_c and

T_c stemming from the formation of ultrafine-grained microstructures in presently-known high- T_c superconductors. Since the upper critical field (H_{c2}) exerts a strong influence on J_c , the effect of microstructure on this parameter has also been studied. While synthesizing samples with fine microstructures, it became necessary to address the problem of phase stabilization by impurities in both Al5 and B1 films.

The importance of "in-situ" composites for Air Force applications was recognized and the critical question of ac losses was addressed. Also some additional work was performed with the general objective of advancing the understanding of high- T_c superconducting materials. Some of the important results of the past year of the program in these various areas are summarized in the following section.

4. ACCOMPLISHMENTS

4.1 Mechanism of J_c Enhancement

4.1.1 NbN

During the course of this program (as discussed in the 1980 Annual Report), very high critical current densities, J_c , have been obtained in certain NbN films having ultrafine-grained columnar-void microstructures. The very high J_c 's were attributed partially to the high upper critical fields of these films. It was hypothesized that these high critical fields resulted from an H_{c3} nucleated at the surface of the columnar grains. Further work was done during this period in order to verify this hypothesis.

If an H_{c3} was indeed nucleated on the surface of the columnar grains in the films, one should also be able to observe it on the film surface, providing that the surface was not degraded by being oxidized. To study this possibility, NbN films were deposited and then covered with a layer of insulating Si-N in a continuous process (i.e., without breaking the vacuum). The magnetic field properties of these layered films were then measured. With the field applied normal to the film surface (parallel to the columnar grains) an enhanced H_{c2} was measured, similar to earlier results. With the field applied parallel to the film, which is the appropriate direction for the H_{c3} on the film surface, no enhancement was observed. In another approach toward resolving the question of H_{c3} , the upper critical field of NbN films was determined at temperatures close to 0K. Because of the very high fields required ($>30T$), pulsed fields were used for these tests. Through the cooperation of Prof. Ø. Fischer of the University of Geneva, results have been obtained which indicate that extrapolated upper critical field values, calculated from low-field data taken near T_c , are higher than the

experimental values. This result agrees with the data on the layered NbN-SiN films in that they both cast doubt on the H_{C3} hypothesis. It now appears probable that the enhancement of H_{C2} measured at temperatures close to T_c is the result of a dimensional effect and is not due to an H_{C3} .

4.1.2 Nb₃Ge

An enhancement of J_c was observed earlier in pulse-laser-annealed Nb-Ge thick films ($\sim 5 \mu\text{m}$). This effect was attributed to flux pinning on second-phase nuclei precipitated by the laser pulse. Since the photon energy deposition in metals occurs in a thin surface layer a more significant J_c -enhancement was expected to occur in thinner films. Consequently the critical current density was determined in a series of Nb-Ge films having various thicknesses ranging from ~ 0.3 to $> 3 \mu\text{m}$. Contrary to expectations the J_c of all of the films was degraded after laser processing. This unexpected result was explained in part by film-substrate interdiffusion effects, and by thin sample damage caused by stripping the stabilizing copper film prior to laser treatment. That interpretation was based on the electron microscopy and energy-dispersive X-ray analysis. The above results were presented at the CEC-ICMC meeting in San Diego, August 1981. It was concluded that further work should concentrate on electron-beam annealing that could be effective in thick films. A different experimental approach using contactless J_c determination from magnetization data was also chosen for future work.

The E-beam annealing was performed on a series of thick Nb-Ge film as a function of the beam energy density, E_p , in the range of 0.4 to over 4 joule/cm², and at reference (substrate) temperatures of 20 and 825°C. The Karlsruhe pulsed electron beam facility (PEBA) made available by Dr. O. Meyer was used. The pulse length was 300 nsec, twenty times longer than the laser pulse length. Internal melting occurred at $E_p \geq 4 \text{ joule/cm}^2$ and resulted in a blow-off of the upper film layer. This indicated that achievement of ultrafine-grained microstructures in rapidly quenched thick films on metallic substrates may not be feasible. At pulse energy densities below 4 joule/cm² a slight J_c -enhancement was observed

in the material used for the published laser annealing study. In some other samples, however, that initially contained a different amount of second phase, no J_c -change was found. Work concentrates now on correlating the pulse length (annealing time), energy, and the second-phase nucleation using X-ray diffraction and electron microscopy. Presentation of the F-beam annealing results is scheduled for the 1982 APS March Meeting.

4.1.3 NbC

High J_c values have been observed earlier in a model system of an ultrafine-grained equiaxial NbC microstructure with well-defined grain and void sizes comparable to the coherence length (see 1980 Annual Report). An X-ray photoelectron spectroscopy (XPS) study was performed to determine the nature of the intergranular interfaces. It was established that several angstroms thick Nb_2O_5 barriers are present at grain boundaries. No lower oxides were found. The results suggest that tunnelling is the controlling mechanism for the supercurrent transfer in this model system.

4.2 Preparation of Ultrafine-Grained Microstructures

The microstructures of laser and e-beam annealed Nb_3Ge films have been observed using scanning transmission electron microscopy (STEM). In films which were laser annealed at a power level of 1.5 joule/cm^2 there was evidence of melting and some recrystallization into an ultrafine-grained microstructure. However this occurred in only a small fraction of the melted material. Subsequent E-beam annealing experiments described in Section 4.1.2 indicated that obtaining such a microstructure in a significant fraction of film volume is probably not feasible.

4.3 The Effect on T_c of Variations in Grain Size

There was no new activity toward this objective. Conclusions stated in previous reports are still considered valid.

4.4 Correlation of Mechanical Load Properties and Microstructure

The mechanical load properties of NbN films have been measured in collaboration with J. Ekin at the National Bureau of Standards, Boulder,

Colorado. The dependence of J_c on uniaxially applied strain was determined at 4.2K in fields of up to 22T. The films were deposited on Hastelloy B substrates. In the whole field range no strain-dependence of J_c was observed until an $\sim 0.7\%$ strain was applied. At this level there was the beginning of a sharp degradation in J_c . This coincided with the yielding of the Hastelloy substrate. Due to the difference in expansion coefficients between film and substrate, there is an $\sim 0.2\%$ compressive strain introduced as a result of cooling from 773 to 4.2K. The results thus indicate that NbN can withstand a tensile strain of at least 0.5% without experiencing degradation in current-carrying capacity.

This result is significantly different from that obtained in Al₅ structure superconductors. In these materials, deterioration in critical current already begins at a strain of $\sim 0.2\%$. A scientific paper in collaboration with Dr. Ekin is in preparation and is scheduled for presentation at the American Physical Society 1982 March Meeting.

4.5 Stabilization of High- T_c Phases

4.5.1 NbN

Based on the conclusion that cubic NbN, when grown at low temperatures ($\sim 500^\circ\text{C}$) is an impurity stabilized phase, a virtual leak was incorporated into the NbN sputtering system to provide a source of oxygen in the substrate area. Following installation of the virtual leak, high- T_c ($\sim 14\text{K}$) NbN (films) were for the first time deposited on a moving metallic tape substrate. In addition, amorphous-recrystallized films with T_c 's of $> 15\text{ K}$ have now also been obtained on metallic substrates. Previously this has been accomplished only on Al₂O₃ substrates. These data reemphasize the conclusion that controlling the impurity level of NbN grown at low temperatures is necessary in order to form the high critical temperature NbN phase.

4.6 AC Losses and Related Topics

An effort was made to develop a method of investigating the electric transport properties of an "in-situ" composite matrix between the

discontinuous filaments. Consequently, a theory of current distribution near the end of an untwisted filamentary superconductor was elaborated. This theory permits one to determine the effective transverse matrix resistivity, σ_{\perp} , from magnetization measurements.

Subsequently, end effects on the loss in a short superconducting strip made of untwisted filaments were calculated. This led to a precise definition of the critical length of a conductor that takes into account the existence of a current domain boundary at each end. Although the calculations apply to a continuous filamentary material a similar domain structure would be expected for other types of superconductors such as the "in situ". One paper on the end effects was published and another accepted for publication.

4.7 Other Topics

In addition to the above, some work has been done on other topics to help fulfill a general objective of the program, i.e., to further the understanding of high- T_c superconductors.

4.7.1 Preparation of Pseudobinary V-Si-C Films by Implantation

To investigate the effect of carbon substitution on T_c of V_3Si , a series of V-Si films ranging in composition from $\sim V_{90}Si_{10}$ to $V_{75}Si_{25}$ were C-implanted with levels of up to 5 at. % (average). This was done by J. M. Williams at the Oak Ridge National Laboratory. These films along with control samples of as-deposited and Ne-implanted films of similar compositions were annealed. After annealing T_c enhancements in each set of films were observed. The consistently largest enhancements, however, were seen in the carbon-implanted films. It is concluded that the increases in T_c are due to a combination of effects. Chemical analyses showed that oxygen had been incorporated into the films during the sputtering process. Part of the T_c enhancement is believed to be due to a mechanism in which the silicon oxide, formed during deposition provides a controlled source of Si which reacts with excess vanadium to form localized regions of nearly stoichiometric V_3Si — when the films are

annealed. This mechanism is operative in all of the films. The significantly higher T_c 's seen in the C-implanted films indicate that the presence of carbon provides an additional enhancing effect. This is probably due to the carbon being incorporated into the A15 lattice thereby forming a V-Si-C pseudobinary compound whose composition is close to the ideal 3/1 stoichiometry. The inability to raise T_c in any of the films above that of pure V_3Si (17.1K) is believed to be associated with a lattice instability which results when the lattice spacing in the A15 structure becomes too small. This type of instability is well documented in Nb-based compounds. Because of the complicating factors produced by the presence of oxygen in the films, the present results cannot be considered definitive with respect to the possibility of producing $V_3(Si,C)$ films with T_c 's greater than that of V_3Si . Further work on oxygen-free films is required.

Some of these data on V-Si-C films were presented at the March 1981 meeting of the American Physical Society. A more complete version is accepted for publication in a book on "Metastable Materials Formation by Ion Implantation."

4.7.2 Ultrasonic Attenuation of NbN Films

The measured attenuation of surface acoustic wave propagating in many thin NbN films with a column-void microstructure is anomalous for the superconducting state. In its temperature dependence, the attenuation deviates markedly from the expected BCS form, at a given temperature being in excess of the BCS value. A simple treatment of a two-dimensional model of a superconductor with Kosterlitz-Thouless vortex-antivortex pairs produces the shape of the attenuation but gives too small a magnitude. A more complete study of a two-dimensional superconductor with strong scattering is in progress. This work is in collaboration with Professor M. Tachiki, Tohoku University, Japan. The experimental work and earlier modeling were done at the University of Wisconsin-Milwaukee (Professor M. Levy) and the Westinghouse R&D Center.

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10. "Possible Ultrasonic Evidence for the Existence of Vortex-Antivortex Pairs," Hans P. Fredricksen, Moises Levy, M. Ashkin and J. R. Gavaler, Accepted for publication in Molecular Crystals and Liquid Crystals.
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12. "Superconducting Critical Temperatures of C-Implanted Al₅ Structure V-Si," J. R. Gavaler, A. I. Braginski, A. S. Manocha, B. R. Appleton, C. W. White and J. M. Williams, Accepted for publication in Metastable Formation by Ion Implantation, S. T. Picraux and W. J. Choyke, Ed., Elsevier North Holland, Ind. (1982).
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6. PERSONNEL CONTRIBUTING TO PROGRAM

M. Ashkin

A. I. Braginski

W. J. Carr, Jr.

J. R. Gavalier

J. Gregg

M. A. Janocko.

7. COUPLING ACTIVITIES

1. "The Effect of Laser Annealing on the Critical Current Density of Nb_3Ge ," A. I. Braginski, J. R. Gvaler, B. R. Appleton and C. W. White, contributed paper presented at the A. P. S. Spring Meeting, Phoenix, AZ (March 1981).
2. "Critical Temperature of Al_5 Structure V-Si-C Films," J. R. Gvaler, A. I. Braginski, B. R. Appleton, C. W. White and J. M. Williams, contributed paper presented at the A. P. S. Spring Meeting, Phoenix, AZ (March 1981).
3. "Workshop on Superconductors for Magnets: Frontiers of Technology," organized and chaired by A. I. Braginski at the 7th International Magnet Conference, Karlsruhe, West Germany, March 30-April 3, 1981.
4. "Superconductors for High Field Magnets," a seminar given by A. I. Braginski at the Grenoble Nuclear Center, France, on May 4, 1981.
5. "AC Losses in 'In-Situ' Composites," a seminar given by A. I. Braginski at the Saclay Nuclear Center, France, on May 7, 1981.
6. "Current Distribution Near the End of a Filamentary Superconductor," a contributed paper presented by W. J. Carr, Jr. at the 1981 Intermag Conference in Grenoble, France, May 15, 1981.
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10. "End Effects on the Loss for Short Filamentary Superconductors," W. J. Carr, Jr., Contributed paper presented at the CEC-ICMC Conf., San Diego, CA, August 1981.
11. "Chemical Vapor Deposition of Superconductors," A. I. Braginski, Invited paper presented at the VIII International CVD Conference, Gouvieux, France, September 1981.
12. "Superconducting Critical Temperatures of Al_5Si ," J. R. Gavaler, A. I. Braginski, A. S. Manocha, B. R. Appleton, C. W. White and J. M. Williams, Contributed paper presented at the Material Research Conf., Boston, MA, November 1981.

8. PATENTS AND INVENTIONS

None.

